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THE NEED FOR EXPERIMENTALLY DETERMINED X-RAY ELASTIC CONSTANTS

by

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THE NEED FOR EXPERIMENTALLY DETERMINED X-RAY ELASTIC CONSTANTS*

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ABSTRACT

In order to convert residual strains measured by x-ray diffraction techniques into residual stresses, appropriate x-ray elastic constants have to be measured. Since these x-ray elastic constants may depend on the metallurgical state, deformation, and entire specimen history, errors in stress values may result if the constants are not measured for representative material states. In the present work, it is shown that in some cases these errors may be large.

The x-ray elastic constant, $S_2/2 = (1 + \nu)/E$, has been measured for the 211 CrK_α reflection from an Armco iron sample which had been previously deformed by rolling (69 pct. reduction in thickness) and for the 211 CrK_α and 310 CoK_α reflections from a 1045 steel specimen which had been previously elongated in tension. The measured elastic constant for the Armco iron specimen was 40 pct. lower than the value calculated from the average of the Reuss and Voigt values.

INTRODUCTION

In order to relate strains measured by x-ray diffraction techniques to a stress value, one must have appropriate values for the elastic constants. As has been pointed out many times in the literature, the elastic constants determined by mechanical means may not be applicable because of features inherent in the x-ray measurement (1-3). The x-ray technique is inherently selective in that the strain deduced from the change in position of an x-ray diffraction peak represents an average value in a given crystallographic direction for only those grains in the polycrystalline aggregate which are oriented to contribute to the particular x-ray reflection. Therefore, the effective values of E (Young's modulus) and ν (Poisson's ratio) in these orientations may differ from the overall average orientation, the latter being measured in a mechanical test.

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The x-ray elastic constants may be obtained theoretically or experimentally. A rigorous theoretical calculation of the x-ray elastic constants requires a complete theoretical solution of the influence of elastic anisotropy and grain interactions on x-ray strain measurements. Because such a solution has not yet been achieved, various assumptions of the nature of the coupling of the crystallites have been used. The most common are those of Voigt (4), which assumes equal strains in all crystallites, Reuss (5), which assumes equal stresses in all crystallites and Kroner's (6,7) "coupled crystallites" model. A common procedure used by a number of workers in this field is to take the arithmetic average of the Reuss and Voigt values calculated for a material with random crystallite orientation. X-ray elastic constants have also been calculated by using one or more of the above assumptions and considering the effects of nonrandom crystal orientation (8,9) and the effects of more than one phase (10,11).

The x-ray elastic constants calculated by any of the methods described above do not always agree with the experimentally determined values. The magnitude of the disagreement depends on the state of the materials and there is evidence that "constant" x-ray elastic constants may not exist. They may depend on composition and second phase components (2,12), grain size (3), microstructure (3), deformation (13-15), and heat treatment (16). The magnitude of these effects depends on the hkl reflection being considered.

The purpose of this paper is to report experimentally determined x-ray elastic constants which differ substantially from calculated values, to discuss their importance, and to discuss possible reasons for the large disagreements between calculated and measured values.

EXPERIMENTAL PROCEDURE

Specimen Preparation and Deformation

The materials employed in this study were Armco iron and 1045 steel. A 0.89 mm (.035 in) thick flat tensile specimen of 1045 steel (designated 1045-5) and a 0.89 mm (.035 in) thick block of Armco iron (designated Armco-9) were prepared in the manner outlined in (17). The final samples (before deformation) were in the annealed, furnace-cooled condition with dimensions also given in (17). The final annealing was done in a vacuum which was at worst 1.3 mPa (10^{-5} torr). Electropolishing was then performed in a phosphoric-sulfuric acid bath (18). Sample 1045-5 was subsequently elongated on an Instron tensile machine at a strain rate of $\approx 2.7 \times 10^{-4}$/sec to a true strain of 13 pct. It was deformed to the ultimate load with a true stress preceding unloading of 706.7 MPa (102,496 psi). Sample Armco-9 was subsequently reduced in thickness 69 pct. (final thickness = .267 mm (0.0105 in)) by rolling on a two-high mill driven at 31 rpm (roll diameter = 133.4 mm (5.25 in)). The reduction in thickness was 0.1 mm (0.004 in) per pass and the sample was reversed end for end after each pass. After the rolling deformation, a tensile specimen with the same dimensions as 1045-5 was carefully machined with excess coolant.

These materials and deformation history were chosen because a previous study (17) showed the rolled Armco iron to possess large oscillations in d_ψ vs. $\sin^2\psi$ (ψ and d_ψ are defined in the next section) and the tensile deformed 1045 steel did not. Therefore, these two specimens should provide information on the usefulness of the calculated x-ray elastic constants for samples which do or do not satisfy the classical linear d_ψ vs. $\sin^2\psi$ requirement.

Measurement of X-Ray Elastic Constants

The basic relation for x-ray stress analysis written for a uniaxial stress state, (surface stress = σ), is (2):

$$\epsilon_\psi = \frac{S_2}{2}\,\sigma\,\sin^2\psi + S_1\sigma = \left(\frac{\Delta d}{d}\right)_\psi = \frac{d_\psi - d_o}{d_o} \qquad (1)$$

where ψ is the angle from the sample normal in the plane defined by the sample normal and the direction of the stress (it is the angle of tilt of the specimen away from the usual diffraction position for which the incident and diffracted beam make equal angles with the sample surface), ϵ_ψ is the strain in the direction defined by ψ, d_ψ is the lattice spacing in the direction defined by ψ, d_o is the lattice spacing in the unstressed state and S_1 and $S_2/2$ are elastic constants given by:

$$S_1 = -\frac{\nu}{E} \quad , \quad \frac{S_2}{2} = \frac{1+\nu}{E} \qquad (2)$$

To measure the x-ray elastic constants, a uniaxial tensile test has to be performed within the elastic range (on the diffractometer). From measurements of d_ψ vs. $\sin^2\psi$ in the plane given by the sample normal and the applied load with different known values of the applied tensile stress $\sigma = \sigma_{app}$, one obtains m* (given by Eq. (3)) and $d_{\psi=0}$ as a function of σ_{app}.

$$m^* \equiv \left(\frac{S_2}{2}\right)\sigma = \frac{\partial \epsilon_\psi}{\partial \sin^2\psi} = \frac{1}{d_o}\,\frac{\partial d_\psi}{\partial \sin^2\psi} \qquad (3)$$

Applying these values in the partial differentiation of Eq. (3) and Eq. (1) written for $\psi = 0$, one obtains the x-ray elastic constants as:

$$\frac{S_2}{2} = \frac{\partial m^*}{\partial \sigma_{app}} \qquad (4)$$

$$S_1 = \frac{1}{d_o}\,\frac{\partial d_{\psi=0}}{\partial \sigma_{app}} \qquad (5)$$

Since d_ψ, $d_{\psi=0}$ and d_o rarely differ by more than 1 pct. (and since d_o in Equations 3-5 is a multiplier and not used in a difference), the value of d_o has been chosen equal to $d_{\psi=0}$ in this work.

All of the x-ray measurements were made with a Picker diffractometer equipped with filtered radiation, a scintillation detector and a pulse-height analyzer set for 90% acceptance of the K_α peak. The peak position at a ψ value other than 0 was measured by the parafocus method (18) in which the receiving slit is moved into the calculated focal point. A fixed vertical slit at the stationary counter assured that the same range of orientations was examined at each position. The ω motion on the diffractometer was employed for the ψ rotation. The method of positioning the sample to within ± .025 mm (± .001") of the center of the goniometer is described in (17-19). The peak position was determined to within ± .005° 2θ by performing a least squares fit to a parabola for 10-20 data points with intensity greater than 85 pct. of the maximum intensity (for each data point the time necessary to accumulate 100,000 counts was measured).

Deformation on the X-Ray Unit

A small tensile unit for use on the diffractometer was constructed and is shown in Figure 1. It can apply a uniaxial load on a tensile specimen but the magnitude of the load had to be determined in some other way. This was done as follows: A strain gage was applied to the back of the tensile bar on which the measurements were to be made. As the specimen was stressed, a value of strain could be recorded. The

Fig. 1. Tensile device for the x-ray diffractometer. (Shown in the vertical position--the results presented in this study were obtained with the load applying part rotated 90° about the normal to the specimen surface.)

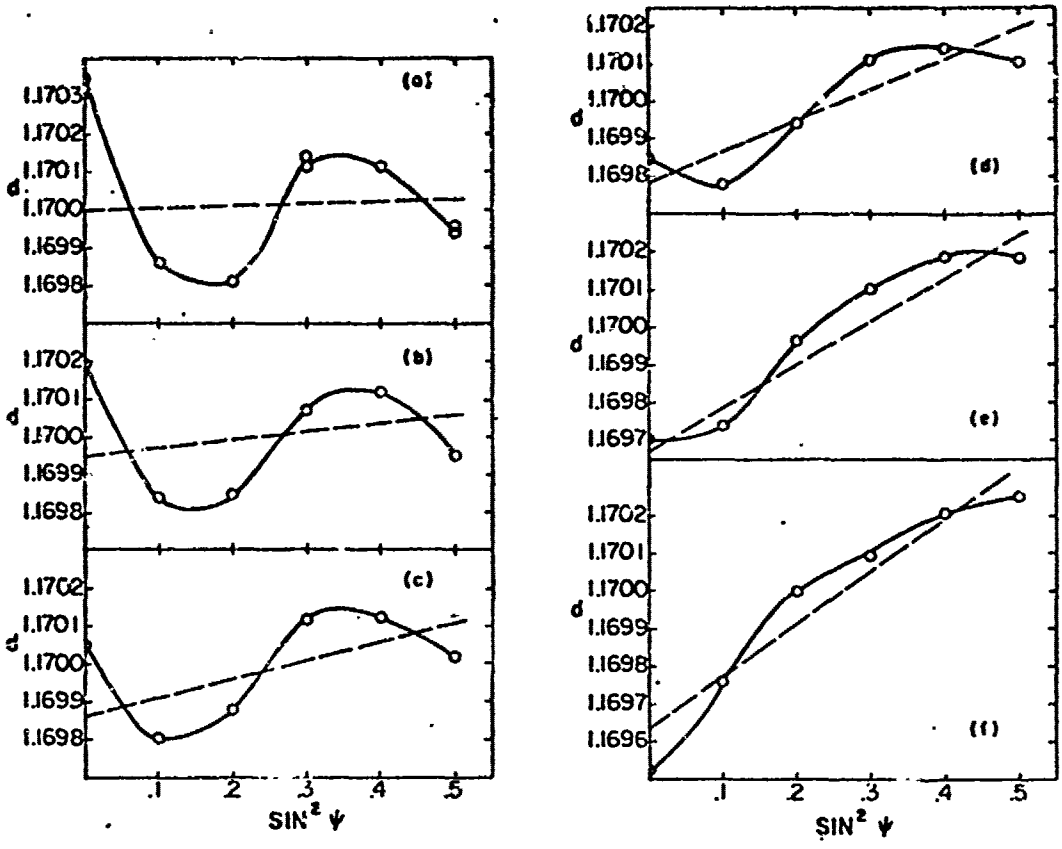

Fig. 2. The dependence of d (or d_ψ) on $\sin^2\psi$ for various applied loads for specimen Armco-9 previously reduced 69 pct. by rolling; 211 peak with CrK_α; —— curve through experimental points; – – – represents slope obtained with the Marion-Cohen method described in Ref. (17). a) $\sigma_{app} = 0$; b) $\sigma_{app} = 46.0$ MPa (6667 psi); c) $\sigma_{app} = 99.8$ MPa (14,476 psi); d) $\sigma_{app} = 191.8$ MPa (27,810 psi); e) $\sigma_{app} = 269.3$ MPa (39,048 psi); f) $\sigma_{app} = 375.7$ MPa (54,476 psi).

load corresponding to this strain was determined by taking the same sample, strain gage and grips and loading it on an Instron testing machine until that value of strain was reached. The track in which the grips slide has been precisely machined to eliminate bending. The maximum bending strain was measured to be 25 μm/m. The gearing at the end of the unit makes it possible to apply the load very easily without deflecting the unit. The tensile unit is mounted so that the load applying rig can be rotated about the normal to the specimen surface. At the base of the support is a micrometer adjustment to allow for accurate specimen positioning.

RESULTS

The dependence of d_ψ on $\sin^2\psi$ for the 211 CrK_α reflection from sample Armco-9 is given as a function of applied load in Figures 2a-2f. A complete discussion of the oscillations in d_ψ vs. $\sin^2\psi$ is given in Ref. (17), where a method is presented for obtaining the true slope (or

m*), independent of the oscillations. This method was used to subtract out the effects of the deviations from linearity and obtain the slopes represented by the dashed lines in the figure. The resulting values of m* can then be plotted vs. σ_{app} as is shown in Figure 3. All of the points lie on a good straight line except for the last point. At this load the sample was apparently no longer behaving in an elastic manner--that is, it was microscopically plastic even though it was still below the macroscopic yield stress of 703.9 MPa (102,095 psi). This is substantiated by the observation that the oscillations in d_ψ vs. $\sin^2\psi$ reversed. Therefore, this point was not included in the x-ray elastic constant determination. A least-squares line was passed through the first five points to obtain the value of $S_2/2$ given in Table I. The standard deviation given is the standard deviation of the slope obtained from the fit. The "texture-independent directions" approach of Hauk et. al. (20) to obtain m* was tried with this data. This method was considered inadequate because it resulted in a nonlinear dependence of m* on σ_{app} and $S_2/2$ determined from a best fit straight line was excessively large.

A value of the other elastic constant, S_1, cannot be obtained from this data because only the slope of the straight line (m*) can be obtained from the techniques described in (17) and not its intercept. Therefore, since $d_{\psi=0}$ cannot be obtained as a function of applied load, S_1 cannot be determined from Eq. (5). This is not a severe limitation, however, because $S_2/2$ is the only elastic constant needed in the commonly used "$\sin^2\psi$-method".

The experimental results for d_ψ vs. $\sin^2\psi$ for sample 1045-5 are given in Figures 4 and 5 for the 211 CrK_α and the 310 CoK_α reflections, respectively. Since this sample had little or no oscillations in d_ψ vs.

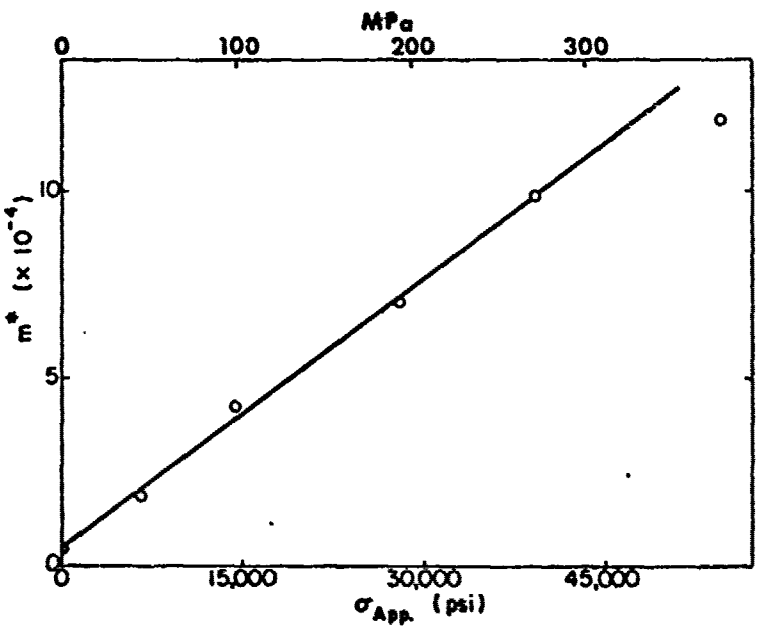

Fig. 3. m* vs. applied load (σ_{app}) for specimen Armco-9 previously reduced 69 pct. by rolling; 211 peak with CrK_α. —— least-squares line through the first five points.

Table I. X-Ray Elastic Constants

	hkl	Units	Experimentally Determined*		Theoretical		
			ARMCO-9, Previously Reduced 69 pct. by Rolling	1045-5, Previously Elongated to a True Strain of 13 pct.	Reuss	Voigt	Average of Reuss and Voigt
$S_2/2$	211	10^{-8} MPa^{-1}	349.5 (± 10.2)	506.1 (± 16.0)	593.1	567.0	580.0
		10^{-8} psi^{-1}	2.41 (± .07)	3.49 (± .11)	4.09	3.91	4.00
	310	10^{-8} MPa^{-1}		723.6 (± 23.2)	904.8	567.0	736.6
		10^{-8} psi^{-1}		4.99 (± .16)	6.24	3.91	5.08
S_1	211	10^{-8} MPa^{-1}		-103.0 (± 7.3)	-117.5	-108.8	-113.1
		10^{-8} psi^{-1}		-0.71 (± .05)	-0.81	-0.75	-0.78
	310	10^{-8} MPa^{-1}		-158.1 (± 10.2)	-221.9	-108.8	-165.3
		10^{-8} psi^{-1}		-1.09 (± .07)	-1.53	-0.75	-1.14

*Numbers in parenthesis are standard deviation.

$\sin^2\psi$, it wasn't necessary to obtain d_ψ at as many ψ values as done previously. Even though the deviations from linearity were small in this sample, the Marion-Cohen method described in (17) was used to obtain m^* because more accurate values could be obtained. A plot of m^* vs. σ_{app} is given in Figure 6 for both 211 and 310 reflections. The lines drawn on the figure are a least-squares fit and from the slope of these lines the values given in Table I were obtained for the elastic constant $S_2/2$.

Since the deviations from linearity were not large in this sample, the other elastic constant, S_1, could be determined because the positioning of the straight line and the determination of the intercept ($d_{\psi=0}$) could be done relatively accurately. A plot of $d_{\psi=0}$ vs. σ_{app} is given in Figure 7 and it can be seen that deviations from a straight line are quite small. A least-squares fit was performed and the x-ray elastic constant, S_1, given in Table I was obtained from the slope of this line (see Eq. (5)).

DISCUSSION AND CONCLUSIONS

The difference between the measured and the theoretical x-ray elastic constants can be seen in Table I. Since the most commonly used theoretical x-ray elastic constants are the average of the Reuss and Voigt values, they will be used in this discussion. The measured x-ray elastic constant, $S_2/2$, for the 211 reflection from the heavily rolled Armco iron sample is 40 pct. lower than the average of the Reuss and Voigt values. The measured constants for the tensile deformed 1045 steel are closer to the calculated values: The experimental $S_2/2$ value for the 211 reflection does not lie within the Reuss and Voigt limits and is 13 pct. lower than the average of the two values; the value for the 310 reflection is only 2 pct. lower than the average calculated

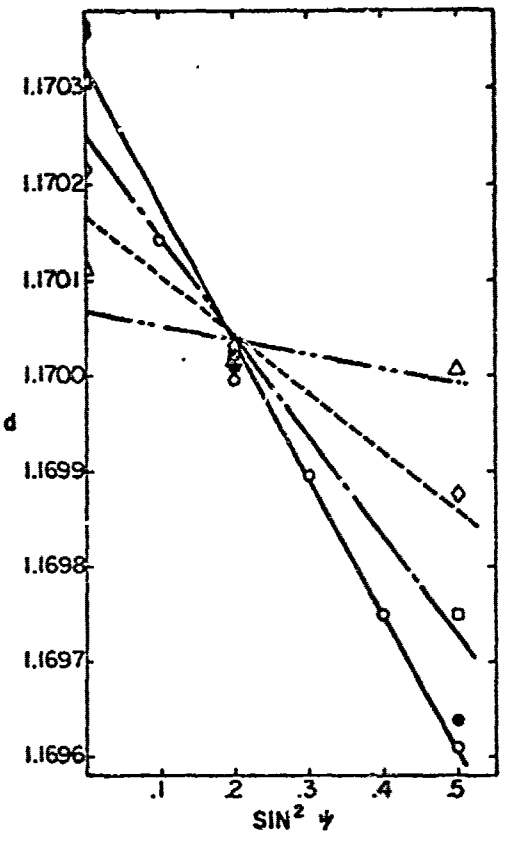

Fig. 4. The dependence of d (or d_ψ) on $\sin^2\psi$ for various applied loads for specimen 1045-5 previously elongated to a true strain of 13 pct.; 211 peak with CrK_α; the straight lines represent the slope obtained with the Marion-Cohen method described in Ref. (17).

● ———————— σ_{app} = 0 (O → Ref. (17))

□ ———— - - ———— σ_{app} = 65.5 MPa (9500 psi)

◇ - - - - - - - - - σ_{app} = 137.9 MPa (20,000 psi)

△ ———— - - ———— σ_{app} = 211.0 MPa (30,600 psi)

value and if the standard deviation is considered, there is no difference. The experimental results for the other x-ray elastic constant, S_1, behave in a manner similar to $S_2/2$.

The 40 pct. difference between the calculated and experimental value of $S_2/2$ for the rolled Armco iron sample may seem large but a number of large changes in the experimentally determined elastic constants have been reported in the literature. S. Taira et. al. (13) have reported a decrease in $S_2/2$ with increasing plastic deformation in iron with 0.01 pct. carbon. At a plastic strain of 20 pct., $S_2/2$ for the 211 reflection had decreased by 36 pct. from its annealed value. Prümmer and Macherauch (14) found a 27 pct. decrease in $S_2/2$ (for the 211 reflection) with deformation for a uniaxially deformed sample of 0.86 pct. C steel. Esquivel (15) has reported a 30-45 pct. decrease

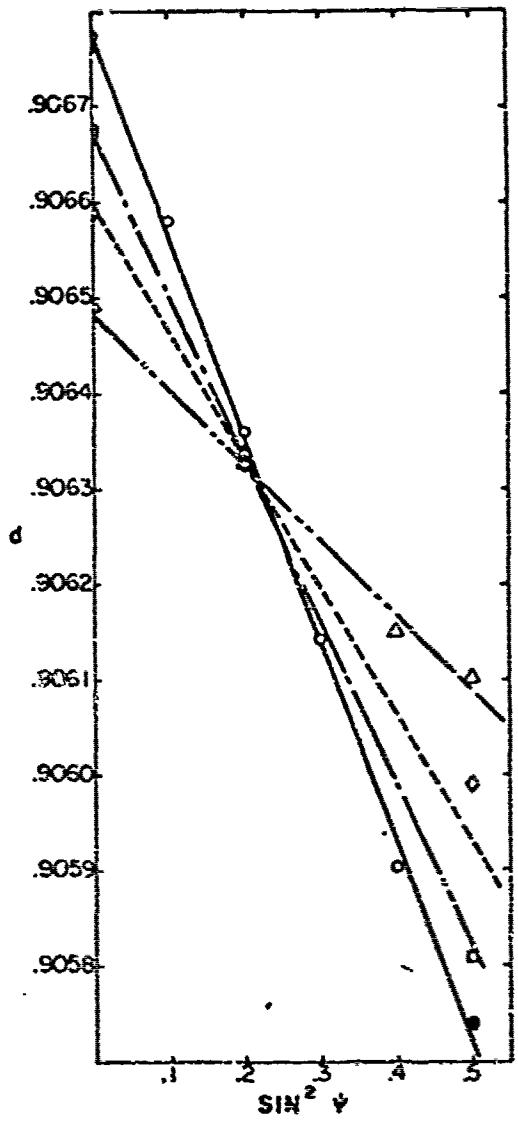

Fig. 5. The dependence of d (or d_ψ) on $\sin^2\psi$ for various applied loads for specimen 1045-5 previously elongated to a true strain of 13 pct.; 310 peak with CoK_α; the various straight lines represent the slope obtained from the Marion-Cohen method described in Ref. (17).

● ———————— σ_{app} = 0 (○ → Ref. (17))

□ ———— - ———— σ_{app} = 59.3 MPa (8,600 psi)

◇ - - - - - - - - - - σ_{app} = 128.3 MPa (18,600 psi)

△ ———— - - ———— σ_{app} = 201.4 MPa (29,200 psi)

in the elastic constants measured after uniaxial plastic deformation for several hardened steels.

The rolled Armco iron sample used in this study was highly textured and probably had a highly deformed microstructure. Therefore, it

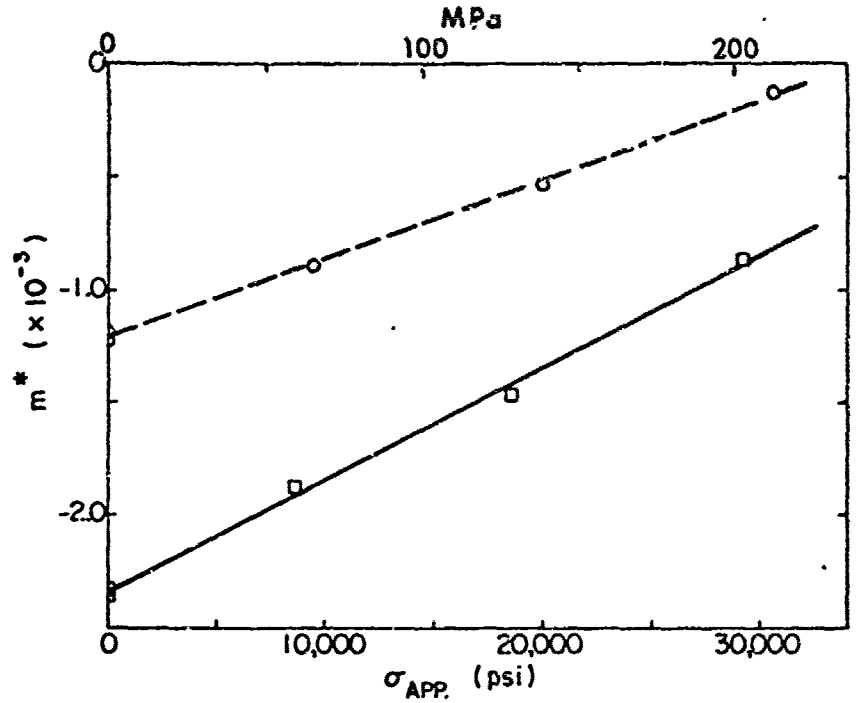

Fig. 6. m^* vs. applied load (σ_{app}) for specimen 1045-5 previously elongated to a true strain of 13 pct.

○ - - - - - - - - 211 peak with CrK_α

□ ———————— 310 peak with CoK_α

is quite likely that the x-ray elastic constants will decrease substantially from their annealed value because only certain regions (the coherently diffracting subgrain interior regions) contribute to the peak and they may be straining in a different manner than in the annealed state. (For a more complete discussion see Ref. (17).) Macherauch and Müller (21) have determined $S_2/2$ for the 211 reflection from Armco iron which had been reduced 75 pct. by cold rolling and subsequently annealed for 4 hours at 500°C. They obtained $S_2/2 = 594.5 \times 10^{-8}$ MPa^{-1} (4.10×10^{-8} in^2/lb). If one assumes that the values are suitable for comparison (similar grain size, etc.), it can be seen that the substructure has probably played a large role in the decrease observed in the results reported here. Additional support for the importance of the microstructural state is given by Fuks and Belozerov (22). They elastically loaded 15-20 μm thick condensed nickel films on the diffractometer and observed that as the size of the coherently diffracting regions increased, $S_2/2$ decreased from 938.8×10^{-8} MPa^{-1} to 591.8×10^{-8} MPa^{-1} for the 400 reflection and remained essentially constant for the 222 reflection. They also found that the anisotropy decreased as the size of the substructural elements increased.

The measured elastic constants for the tensile deformed 1045 steel are similar to those reported in the literature (2). The reason that

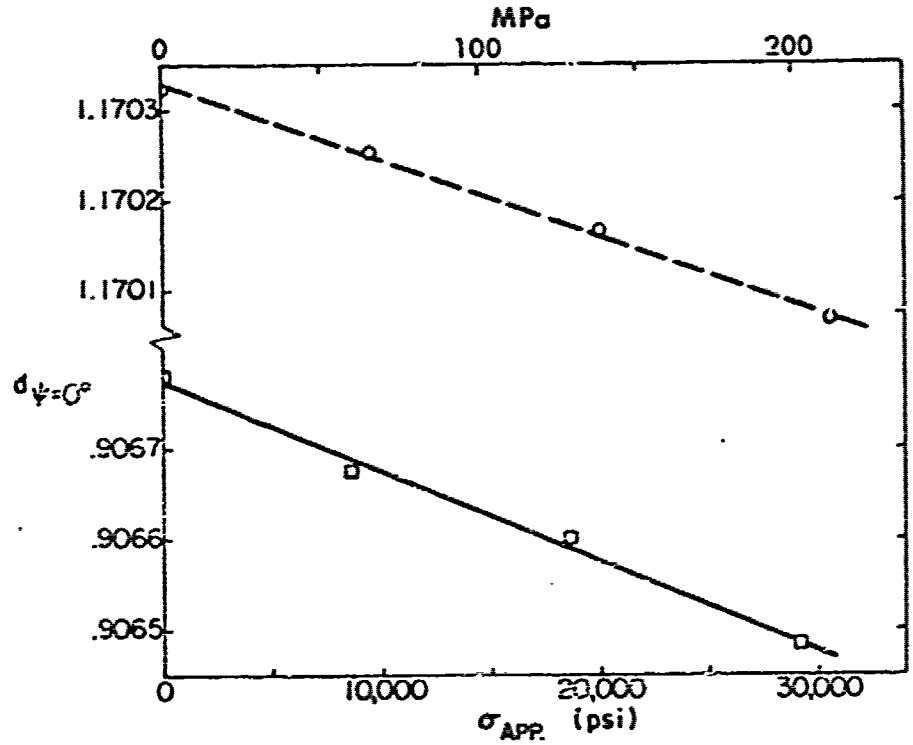

Fig. 7. $d_{\psi=0}$ vs. applied load (σ_{app}) for specimen 1045-5 previously elongated to a true strain of 13 pct.

○ - - - - - - - - - - 211 peak with CrK_α

□ ———————— 310 peak with CoK_α

$S_2/2$ for the 211 reflection is farther below the average of the Reuss and Voigt values than the 310 value is probably because the 211 direction is a "softer" direction than the 310 direction and is affected more by deformation. This is substantiated by the experimental observation that residual strains measured with the 310 reflection are always higher than those measured with the 211 reflection (2,17).

Based on the results presented here, it is obvious that if one is measuring residual stresses which originate with deformation, care should be taken to use appropriate x-ray elastic constants. As mentioned previously, additional results in the literature demonstrate large potential errors due to other causes (Prümmer (16) has reported a 25-53 pct. difference between the experimentally measured x-ray elastic constants for the 211 reflection of hardened and annealed steels of the same composition; and, in plain carbon steels $S_2/2$ for the 211 reflection increases 30 pct. as the carbon increases from 0.03 pct. C to 1.0 pct. C (2).) All this experimental evidence demonstrates the necessity of having the experimental x-ray elastic constants for a specimen exactly the same (same composition, grain size, heat treatment, deformation history) as the material being studied. To date, theoretical calculations have not been able to explain these experimental results and are

therefore considered to be inaccurate. Residual stress errors as large as 50 pct. may result if the wrong x-ray elastic constants are used.

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13. ABSTRACT

In order to convert residual strains measured by X-ray diffraction techniques into residual stresses, appropriate X-ray elastic constants have to be measured. Since these X-ray elastic constants may depend on the metallurgical state, deformation, and entire specimen history, errors in stress values may result if the constants are not measured for representative material states. In the present work, it is shown that in some cases these errors may be large.

The X-ray elastic constant, $S_2/2 = (1+\nu)/E$, has been measured for the 211 CrKα reflection from an Armco iron sample which had been previously deformed by rolling (69 pct. reduction in thickness) and for the 211 CrKα and 310 CoKα reflections from a 1045 steel specimen which had been previously elongated in tension. The measured elastic constant for the Armco iron specimen was 40 pct. lower than the value calculated from the average of the Reuss and Voigt values.

Cr K sub alpha (S sub 2/2 nu Cr K sub alpha Co K sub alpha

DD FORM 1 NOV 68 1473 (PAGE 1)

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